## **PCT**

# WORLD INTELLECTUAL PROPERTY ORGANIZATION International Bureau



#### INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification <sup>6</sup> :		(11) International Publication Number: WO 99/58758
D06M 13/127	A1	(43) International Publication Date: 18 November 1999 (18.11.99)
<ul> <li>(21) International Application Number: PCT/US</li> <li>(22) International Filing Date: 12 May 1998 (</li> <li>(30) Priority Data: 09/075,334 11 May 1998 (11.05.98)</li> <li>(71) Applicant: AMERICAN LAUNDRY MACINC. [US/US]; 5050 Section Avenue, Cincing 45212-2099 (US).</li> <li>(72) Inventor: PAYET, George, L.; 7781 Gwenwyn Drive nati, OII 45236 (US).</li> <li>(74) Agents: KENNEY, J., Ernest et al.; Bacon &amp; Thomas, 625 Slaters Lane, Alexandria, VA 22314 (US).</li> </ul>	12.05.9  HINER nati, C	BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GE, GH, GM, GW, HU, ID, IL, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, UZ, VN, YU, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG).  Published  With international search report.

#### (54) Title: DURABLE PRESS/WRINKLE-FREE PROCESS

#### (57) Abstract

Cellulosic fiber-containing fabrics are made wrinkle resistant by a durable press wrinkle-free process which comprises treating a cellulosic fiber-containing fabric with formaldehyde, a catalyst capable of catalyzing the cross-linking reaction between the formaldehyde and cellulose and a silicone elastomer, heat-curing the treated cellulose fiber-containing fabric, preferably having a moisture content of more than 20 % by weight, under conditions at which formaldehyde reacts with cellulose in the presence of the catalyst without a substantial loss of termaldehyde before the reaction of the fermaldehyde with cellulose to improve the wrinkle resistance of the fabric in the presence of a silicone elastomeric softener to provide higher wrinkle resistance, and better tear strength after washing, with less treatment.

#### FOR THE PURPOSES OF INFORMATION ONLY

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

AL	Albania	ES	Spain	LS	Lesotho	SI	Slovenia
AM	Armenia	FI	Finland	LT	Lithuania	SK	Slovakia
AT	Austria	FR	France	LU	Luxembourg	SN	Senegal
ΑU	Australia	GA	Gabon	LV	Latvia	SZ	Swaziland
AZ	Azerbaijan	GB	United Kingdom	MC	Monaco	TD	Chad
BA	Bosnia and Herzegovina	GE	Georgia	MD	Republic of Moldova	TG	T'ogo
BB	Barbados	GH	Ghana	MG	Madagascar	ТJ	Tajikistan
BE	Belgium	GN	Guinea	MK	The former Yugoslav	TM	Turkmenistan
BF	Burkina Faso	GR	Greece		Republic of Macedonia	TR	Turkey
BG	Bulgaria	HU	Hungary	ML	Mali	TT	Trinidad and Tobago
$_{\mathrm{BJ}}$	Benin	ΙE	Ireland	MN	Mongolia	UA	Ukraine
BR	Brazil	IL	Israel	MR	Mauritania	UG	Uganda
BY	Bellanus	18	Iceland	MW	Malawi	US	United States of America
CA	Canada	1T	Italy	MX	Mexico	UZ	Uzbekistan
CF	Central African Republic	JP	Japan	NE	Niger	VN	Viet Nam
$\mathbf{C}\mathbf{G}$	Congo	KE	Kenya	NL	Netherlands	YU	Yugoslavia
CH	Switze:land	KG	Kyrgyzstan	NO	Norway	ZW	Zimbabwe
CI	Côte d'Ivoire	KP	Democratic People's	NZ	New Zealand		
CM	Cameroon		Republic of Korea	PL	Poland		
CN	China	KR	Republic of Korea	PΤ	Portugal		
CU	Cuba	KZ	Kazakstan	RO	Romania		
CZ	Czech Republic	LC	Saint Lucia	RU	Russian Federation		
DE	Germany	LI	Liechtenstein	SD	Sudan		
DK	Denmark	LK	Sri Lanka	SE	Sweden		
EE	Estonia	LR	Liberia	SG	Singapore		

#### DURABLE PRESS/WRINKLE-FREE PROCESS

#### CROSS REFERENCE TO RELATED APPLICATION

This application claims benefit under 35 USC 119(e) of prior pending application 60/046,298 filed May 13, 1997.

10

5

## BACKGROUND OF THE INVENTION

#### Field of invention

15

This invention relates to a durable press/wrinkle-free process for cellulosic fiber-containing fabrics and more particularly to a process which permits high treatment level amounts of formaldehyde and catalysts to impart wrinkle resistance to the cellulosic fiber-containing fabrics while reducing the loss in both tensile and tear strength normally associated with such treatment processes.

## Description of related art

25

30

20

There are a number of known process for treating cellulosic fiber-containing fabrics, such as cotton-containing fabrics, to make them wrinkle-free. These treatment processes include resin or polymer treatment of the fabric, but these are costly and unsatisfactory. Another process for treating cellulosic fiber-containing products relies on formaldehyde to provide durable crosslinking of the cellulose molecules and to thereby impart durable crease

resistant and smooth drying characteristics to these products. However, problems have been encountered with the known processes. A simple, reproducible, completely satisfactory low-cost formaldehyde durable press process has not yet been achieved.

It has long been known to treat cellulosic materials with formaldehyde, as is evidenced by U.S. Patent Number 2,243,765. This patent describes a process for treating cellulose with an aqueous solution of formaldehyde containing a small proportion of an acid catalyst under such conditions of time and temperature that the reaction is allowed to approach its equilibrium. It is further stated that, in carrying out this process, the proportion of the solution of formaldehyde to the cellulose must be at least such that the cellulose is always in a fully swollen state. It is also stated that the time and temperature of the treatment with the solution of formaldehyde and acid catalyst will vary with one another, the time required increasing rapidly as the temperature diminishes. When it is desired, the product may be isolated by washing and drying; preferably at a temperature of about 212°F. The products obtained according to this process are said to show no increase in wet strength and possess a high water imbibition, an increased resistance to creasing and a slight increase in affinity to some direct dyes.

In recent years additional methods have been devised for treating cellulosic fiber-containing products in order to impart durable crease retention, wrinkle resistance and smooth drying characteristics to these products. As discussed, formaldehyde has been crosslinked with cellulose materials to produce these products. It is also known to treat cellulose materials with resins or precondensates of the urea-formaldehyde or substituted urea-formaldehyde type to produce a resin treated durable press product. As noted in U.S. patent Number 3,841,832, while formaldehyde has made a significant contribution to the cotton finishing art, the result has been far from perfect. For instance, in some cases the formaldehyde crosslinking treatment has tended to lack reproducibility, since control of the

formaldehyde cross-linking reaction has been difficult. As noted in United States patent 4, 396,390, lack of reproducibility is especially true on a commercial scale.

Moreover, unacceptable loss of fabric strength has also been observed in many of the proposed aqueous formaldehyde treatment processes. When high curing temperatures were used with an acid or potential acid catalyst, excess reaction and degradation of the cotton often happened which considerably impaired its strength. On the other hand, when attempts were made to achieve reproducibility at temperatures of 106°F or less, much longer reaction or finishing times were usually required, rendering the process economically relatively unattractive. A solution to this is set forth in United States Patent 4,108,598, the entire disclosure of which is herein incorporated by reference.

10

#### SUMMARY OF THE INVENTION

In accordance with the present invention it is possible to obtain good durable press properties in a cellulosic fiber-containing fabric with good strength retention with a process that produces consistent results. This invention relates to a durable press/wrinkle-free process for cellulosic fiber-containing fabrics and more particularly to a process which utilizes formaldehyde and catalysts with silicone elastomers to impart wrinkle resistance to the cellulosic fiber-containing fabrics while reducing loss in both tensile and tear strength. This process is particularly effective on 100% cotton fabric.

#### DESCRIPTION OF PREFERRED EMBODIMENTS

15

20

25

30

10

5

Such cellulosic fiber-containing fabrics include cloth made of cotton or cotton blends. There is a constant consumer demand for better treatment, that is, a more wrinkle-free product and for higher amounts of cotton in the blended fabric, or preferably, a 100% cotton fabric. There is a great demand for a wrinkle-free fabric made entirely of cotton and having good tensile and tear strength. This has been achieved and 100% cotton fabrics are treated today, but only in heavier weight pants or bottom weight fabrics. Unfortunately, the more wrinkle-free the cellulosic containing fabric is made by treatment in a formaldehyde system, the greater the loss in tear and tensile strength.

That is, as the amount of chemicals used in the treating process are increased to obtain an acceptable wrinkle resistance in the treated fabric, the loss in tear and tensile strength fall to unacceptable levels. Polyester fibers are most often blended into the cotton to form a polyester cotton blend fabric to compensate for the loss in strength of the treated cotton. Polyester in

amounts of up to 65% are commonly used. Because of the presence of polyester fibers or other synthetic fibers in the blend, these blended fabrics are sufficiently strong but do not have the comfort or feel of fabrics containing a higher amount of cotton, or most desirably, 100% cotton. The process of the present invention overcomes the disadvantages of the prior art processes and permits the presence of higher percentages of cotton in the blend and even the treatment of lighter weight or shirting weight 100% cotton fabrics to a commercially acceptable wrinkle free standard while retaining adequate strength in the fabric to also make it commercially acceptable. Commercial acceptability of the treated fabric is the ultimate goal of the process.

The durable press process of the present invention for treating cotton containing fabrics and 100% cotton fabric, comprises treating a cellulosic fiber-containing fabric with aqueous formaldehyde and a catalyst capable of catalyzing the crosslinking reaction between formaldehyde and cellulose in the presence of a silicone elastomer, heat curing the treated cellulosic fiber-containing fabric, preferably having a moisture content of more than 20% by weight, under conditions at which formaldehyde reacts with the cellulose in the presence of a catalyst and without the substantial loss of formaldehyde before the reaction of formaldehyde with cellulose to improve the wrinkle resistance of the fabric while reducing the loss in both tensile and tear strength. It is preferable that the cellulose containing fabric is in the fully swollen state.

Any silicone eslastomer may be used in the present invention. Silicone elastomers are known materials. Silicone elastomers have a backbone made of silicon and oxygen with organic substituents attached to silicon atoms comprising n repeating units of the general formula:

The groups R and R¹ may be the same or different and includes for example, lower alkyl, such as methyl, ethyl, propyl, phenyl or any of these groups substituted by hydroxy groups, fluoride atoms or amino groups; in other words, reactive groups to cellulose.

The silicones used to make the silicone elastomers in the present invention are made by conventional processes which may include the condensation of hydroxy organosilicon compounds formed by hydrolysis of organosilicon halides. The required halide can be prepared by a direct reaction between a silicon halide and a Grignard reagent. Alternate methods may be based on the reaction of a silane with unsatutrated compounds such as ethylene or acetylene. After separation of the reaction products by distillation, organosilicon halides may be polymerized by carefully controlled hydrolysis to provide the silicone polymers useful in the present invention.

For example, elastomers may be made by polymerization of the purified tertramer using alkaline catalysts at 212-302 degrees F., the molecular weight being controlled by using a monofunctional silane. Curing characteristics and properties may be varied over a wide range by replacing some methyl groups by -H, -OH, fluoroalkly, alkoxy or vinyl groups and by compounding with fillers as would be appreciated by one of ordinary skill in the art.

Silicone elastomers used in the present invention are high weight materials, generally composed of dimethyl silicone units (monomers) linked together in a linear chain. These materials usually contain a peroxide type catalyst which causes a linking between adjacent methyl groups in the form of methylene bridges. The presence of crosslinking greatly improves the durability of the silicone elastomer on cellulose by producing larger molecules.

5

10

15

20

It is also possible to produce a reactive silicone elastomer, which is one where reactive groups capable of reacting with the substrate have been added to the linear dimethyl silicone polymer. These silicones are capable of reacting both with cellulose substrates as well as with most protein fibers, and are characterized by much greater durability of the silicone polymer on the substrate, even approaching the life of the substrate.

5

10

15

20

25

30

Therefore silicone elastomers which give off reaction gases or chemicals indicating chemical reaction with the substrate are much preferred over non reactive silicone elastomer, but this is not to say that non reactive silicone elastomers cannot be used in the process. Different elastomers, by different manufacturers have all shown increases in tensile as well as tear strength, as shown in Tables I and II included herein. Elastomeric silicone polymers have been found to increase strength whereas simple emulsified silicone oils (or lubricants) do not give increases in tensile strength.

The aqueous system containing formaldehyde, an acid catalyst, silicone elastomer and a wetting agent may be padded on the fabric to be treated, preferably to insure a moisture content of more than 20% by weight on the fabric, and then the fabric cured. The padding technique is conventional to the art and generally comprises running the fabric through the aqueous solution which is then passed through squeezing rollers to provide a wet pick-up of about 66%. As is conventional in the art, the concentration of the reactants in the aqueous solution are adjusted to provide the desired amount of reactants on the weight of the fabric (OWF).

It is possible to use unexpected high temperatures which allow the crosslinking reaction to take place before the loss of formaldehyde is great enough to affect the process and provide inadequate treatment. In accordance with this aspect of the invention, the padded fabric may be immediately plunged into a heating chamber at from about 300 to about 325°F. This is an important commercial aspect of the invention as it enables

continuous processing on a commercial scale at speeds of 100-200 yards per minute. It must be appreciated, that this process is designed for commercial applications which are demanding in that the process must be commercially viable.

5

10

15

20

25

30

This may also be accomplished by curing at a low temperature with an active catalyst. It is also possible to use any combination of techniques which prevent the substantial loss of formaldehyde during the curing. For example, a low temperature may be used in combination with an aqueous formaldehyde solution. It would also be possible to use a pressurized system wherein the pressure is greater than atmospheric, thereby preventing the substantial loss of formaldehyde before the formaldehyde crosslinks with the cellulosic fiber-containing fabric being treated.

In addition the process of the present invention uses less formaldehyde than other known processes. Shirting fabrics treated in accordance with the process of the present invention contain approximately 1000 ppm after treatment before steaming on a shirting fabric as compared to 3000 ppm+ by another crosslinking process on a similar shirting fabric. Tests have shown that continuously running steaming chambers to which the treated fabric is exposed should effectively remove residual formaldehyde to concentrations as low as 200 ppm. This is also an important aspect of the present invention in view of consumers concern about the presence of formaldehyde in their purchased garments. It is also possible to wash fabrics either continuously or in batch washers. Both approaches remove essentially all of the formaldehyde.

It is known to add to the fabric a polymeric resinous additive that is capable of forming soft film. For example, such additives may be a latex or fine aqueous dispersion of polyethylene, various alkyl acrylate polymers, acrylonitrile-butadiene copolymers, deacetylated ethylene-vinyl acetate copolymers, polyurethanes and the like. Such additives are well known to the art and are generally commercially available in concentrated aqueous

latex form. Such a latex is diluted to provide about 1 to 3% polymer solids in the aqueous catalyst-containing padding bath before the fabric is treated therewith. One known softener which was virtually the softener of choice in the durable press process using resin treatment or formaldehyde crosslinking was high density polyethylene, Mykon HD. It has been unexpectedly discovered that the substitution of a silicone elastomer for high density polyethylene significantly reduces the loss in tear strength of the treated fabric after washing as well as providing better control of the process as may be seen from the examples. The importance of good control of the process is essential to a commercially viable process to provide a consistent product from run to run which is not adversely affected by variations in atmospheric pressure, humidity and the like.

10

15

20

25

As the cellulosic fiber-containing fabric which may be treated by the present process there can be employed various natural cellulosic fibers and mixtures thereof, such as cotton and jute, Other fibers which may be used in blends with one or more of the above-mentioned cellulosic fibers are, for example, polyamides (e.g., nylons), polyesters, acrylics (e.g., polyacrylonitrile), polyolefins, polyvinyl chloride, and polyvinylidene chloride. Such blends preferably include at least 35 to 40% by weight, and most preferably at least 50 to 60% by weight, of cotton or natural cellulose fibers.

The fabric may be a resinated material but preferably it is unresinated; it may be knit, woven, non-woven, or otherwise constructed. After processing, the formed wrinkle resistant fabric will maintain the desired configuration substantially for the life of the fabric. In addition, the fabric will have an excellent wash appearance even after repeated washings.

This invention is not dependent upon the limited amounts of moisture to control the crosslinking reaction since the crosslinking reaction is most efficient in the most highly swollen state of the cellulose fiber. Lesser amounts of moisture may be used but are less preferred.

However, the silicone elastomer must be present in a sufficient amount to reduce the loss of tensile and tear strength in the fabric normally associated with the treatment of the same fabric in a prior art treatment process which may include the use of softeners such as Mykon HD. The formulation and process of the present invention may be adjusted to meet specific commercial requirements for the treated fabric. For example, formaldehyde and the catalyst concentration may be increased to provide better treatment; then the concentration of the softener is also increased to combat the loss of tear strength caused by the increased amount of catalyst used in the process. This lends itself to computerized control of the systems for treating various fabrics and allows variation in the treatment of different fabrics, which is another advantage of the process of the present invention.

While silicone oils are known as silicone softeners and have found some use in fabric treatment, they suffer serious disadvantages in having a strong tendency to produce non-removable spots. However, the particular silicone elastomer used in the process of the present invention completely overcomes these problems.

Blended fabrics to be treated in accordance with the present invention are immersed in a solution to provide a pick up or on the weight of fabric (OWF) of about 3 % formaldehyde, 1 % of catalyst, 1% of the silicone elastomer. This requires a pickup of about 66% by weight of the aqueous formulation to achieve the above stated percentage of reactants on the fabric. However, when treating 100% cotton fabric chemical concentrations must be increased so that 5% formaldehyde OWF, about 2% catalyst and about 2% elastomer padded onto the fabric. This is contrary to the prior art attempts to treat 100% cotton where the concentration of reactants were decreased because of the loss of strength due to the treatment process. The curing temperature may be about 300° F. In fact, the padded fabric may be plunged into a oven or heating chamber at 300°F.

The silicone elastomer was the commercially available softener Sedgefield Elastomer Softener ELS, which is added as an opaque white liquid which contains from 24-26% silicone, has a pH of from 5.0-7.0 and is readily dilutable with water. When used in the present invention, this product produced DP values at catalyst concentrations of 0.8%, whereas with the Mykon HD, a catalyst concentration of 2.0% was required to give a DP value of 3.50 after 1 washing and 3.25 after 5 washings.

The tensile strength with a catalyst concentration of 0.8% and tear strength are significantly and unexpectedly higher than the 2.0% catalyst required with Mykon HD to give equal DP results. Catalyst concentration of 1.0% ELS is recommended to ensure a margin of safety, such that any variation in treatment will be well within accepted specifications.

The following examples are being presented not as limitations but to illustrate and provide a better understanding of the invention. In order to confirm the fact that formaldehyde was being lost from the conventional processes, experiments were conducted in which the fabric was heated very quickly by very hot air as in the conventional processes as well as in accordance with the present invention.

#### 20 Example 1

5

10

15

As indicated, it is possible to cure with a high enough temperature that the crosslinking reaction is achieved before sufficient formaldehyde is lost preventing good treatment. In this experiment, 100% cotton oxford shirting was padded with formaldehyde (37%) at a concentration of 5.0% OWF, 0.8% OWF of Freecat #9 Accelerator manufactured by Freedom Textile Chemicals Co. and 1.5% OWF of a silicone elastomeric softener, Sedgesoft ELS manufactured by Sedgefield Specialties, to a pickup of approximately 60-70%. The sample was then dried and cured while under tension in an air circulating oven set at 300°F, for 10 minutes.

#### Example 2

Another sample of the same fabric as used in Example 1 was padded with a similar solution differing only in that the catalyst Accelerator #9 was 1.0% OWF. Otherwise the sample was treated precisely the same.

5

#### Example 3

Another sample of the same fabric as used in Example 1 was padded with a similar solution differing only in that the catalyst Accelerator #9 was 2.0% OWF. Otherwise the sample was treated precisely the same.

10

15

20

25

#### Example 4

Another sample of the same fabric as used in Example 1 was padded with a similar solution differing only in that the catalyst Accelerator #9 was 0.4% OWF, and Mykon HD was substituted for the Sedgesoft ELS elastomeric Softener. Otherwise the sample was treated precisely the same.

#### Example 5

Another sample of the same fabric as used in Example 1 was padded with a similar solution differing only in that the catalyst Accelerator #9 was 0.8% OWF, and Mykon HD was substituted for the Sedgesoft ELS elastomeric Softener. Otherwise the sample was treated precisely the same.

#### Example 6

Another sample of the same fabric as used in Example 1 was padded with a similar solution differing only in that the catalyst Accelerator #9 was 1.0% OWF, and Mykon HD was substituted for the Sedgesoft ELS elastomeric Softener. Otherwise the sample was treated precisely the same.

#### Example 7

Another sample of the same fabric as used in Example 1 was padded with a similar solution differing only in that the catalyst Accelerator #9 was 1.5% OWF, and Mykon HD was substituted for the Sedgesoft ELS elastomeric Softener. Otherwise the sample was treated precisely the same.

#### Example 8

5

10

Another sample of the same fabric as used in Example 1 was padded with a similar solution differing only in that the catalyst Accelerator #9 was 2.0% OWF, and Mykon HD was substituted for the Sedgesoft ELS elastomeric Softener. Otherwise the sample was treated precisely the same.

#### Example 9

A sample of the same fabric was washed in a home washer and tumble tried, but not treated with any crosslinking process.

#### Example 10

Another sample of the same fabric served as an untreated, unwashed control.

TABLE NO. I

Sedgefield Silicone Elastomeric Softener ELS vs. MykonHD, High Density Polyethylene

Sample Treatment Date: May 1996

Fabric: New Cherokee 100% Cotton Oxford Shirting

									0				
Example	Fabric	СН,О	Cat # 9	Softener	Amount	Cure Temp.	Cure	Tensile	Tear	Shrink	å	Shrink	ď
ç	Type	%OWF	%OWF		%OWF	ų.	Time Min	WXF	WXF	1 Wash	1 Wash	5 Washes	5 Washes
										WXF%		WXF%	
-	Ordord	5.0	9.0	ELS	1.5	300	ō	45.3 X 46 0	594×452	108 X 0 58	3 50	1 50 X 0 83	3.50
2	Oxford	5.0	10	ELS	1.5	300	10	43.7 X 41.3	48 5 X 42 9	075 X 0 58	3 50	125 X 0 67	3.50
•													
3	Oxford	5.0	2.0	ELS	1.5	300	10	30.0 X 29.0	28 9 X 25 5	78 0 X 27 0	3.50	0 92 X 0 75	350
4	Oxford	5.0	0.4	Mykon HD	1.5	300	10	81.8 X 69 8	103 8 X 79.5	2 00 X 1.42	2.0	250 X 1 08	2 00
2	Oxford	5.0	0.8	Mykon HD	1.5	300	10	53 0 X 58 2	72.9 X 53.4	1 67 X 1 08	2.75	183 X 0 92	2.50
9	Oxford	5.0	1.0	Mykon HD	1.5	300	Q.	47.2 X 47.2	60 3 X 42 4	1.17 X 0.83	3 25	117 X 0 87	2 50
,	Oxford	5.0	1.5	Mykon HD	1.5	300	ç	39.3 X 37 5	36 8 X 26 6	290×880	3 25	075 X 0 33	3 00
8	Oxford	5.0	2.0	Mykon HD	1.5	300	10	34.7 X 35 0	27.8 X 25.5	0.75 X 0.67	3.50	075 X 0 42	325
_6	Control	ł	ı	ı	Į	1	ı	74 3 X 99 0	120.1 X 133.2	2 00 X 1 58	× 1.0	4.42 × 1.83	410
	Umwashed												
10	Control Washed	1	ı	ı	1	_	1	71.7 X 100 B	35.7 X 63.9	ı			

It is clear in Table No. I that samples treated with the elastomeric softener produced higher degrees of durable press than any of the samples treated with Mykon HD. Tensile Strengths are similar as is shrinkage for each degree of treatment.

5

In another experiment, the results shown in Table No. II, samples of 100% cotton oxford shirting were padded with two concentrations of formaldehyde 3.0 and 5.0% OWF, each concentration also treated with three concentrations of Accelerator #9 Catalyst, 0.8, 1.0, and 2.0%. In one half of the samples, Sedgesoft ELS was applied and in the other half Mykon HD was used as the softener. Both softeners were applied at 1.5% OWF. Each of the samples were padded with the respective solutions shown in Table No. II, then cured at 300°F. for 10 minutes under tension. All samples were treated in precisely the same way, intervals were timed.

15

20

10

It is clearly seen in Table II (Example 11 to Example 22 and the control) that after 5 washes, the Sedgesoft ELS samples have almost twice the tear strength of the Mykon HD samples without exception. In addition, again seen, the DP values are higher indicating better smoothness.

TABLE NO. II
Comparison of Softeners, Sedgesoft ELS vs. Mykon HD

Silicone Polymer Emulsion Polyethylene Emulsion

Sedgesoft ELS:

Treatment:

Mykon HD:

Tensile, Filling: 25 lbs.; Tear, Filling: 24 oz

Specification Strength:

54.2 X 66.8 42.9 X 40.6 29.4 X 32.3 27.8 X 29.8 19.6 X 19.9 50.3 X 65.5 43.3 X 58.0 28.7 X 33.2 28.7 X 42.9 5 Washes W X F 26.6 X 24.1 29.8 X 37.7 Tear<sup>2</sup> 5 Washes W X F 56.8 X 65.8 35.5 X 39.8 30.0 X 37.3 47 0 X 53.2 54 0 X 80 0 47.0 X 59.5 38.0 X 51.8 49.7 X 67.5 342×34.5 52.2 X 60.0 Tensile<sup>2</sup> 5 Washes W X F 49.3 X 52.0 5 Washes 5 Washes 7 00 3.00 2.75 3.00 5.90 2.50 2.90 38 3.25 2.75 38 6 2 92 X 2.00 1.67 X 1.33 3.50 X 1.75 2.500 X 1.87 1,75 X 1,42 2.50 X 1.75 2.42 X 1.33 1.91 X 1.00 0.75 X 0.75 2.58 X 1.87 2.00 X 1.33 Shrink 5 Wesh W X F % 1 Wash 2.50 1 Wash 2.75 3.8 2.75 3.25 3.25 3.00 3.25 2.50 2.75 3.00 ይ 2 DO X 158 1.17 X 1.255 1.58 X 1.08 1.67 X 1.58 1.83 X 1.42 1.25 X 1.17 1 92 X 1.25 1.08 X 0.92 2.50 X 1.42 1.75 X 1.17 2.00 X 1.67 Shrink 1 Wash W X F % 75.2 X 50.8 40.0 X 31.8 29.4 X 23.3 27.5 X 21.0 56.4 X 35.4 40 6 X 30.5 63 2 X 43.6 66.2 X 49.0 44.0 X 36.6 60.9 X 41.1 28 6 X 27.5 V X F 38.2 X 34.2 48.7 X 44.0 51.5 X 49.0 44.0 X 48.0 54.8 X 55.7 30 8 X 27.3 43.7 X 39.7 43.2 X 38.2 31.8 X 29.3 49.7 X 48.7 51.8 X 53.3 Tensie Lbs. WXF Cure/Time F./Min. Cure/Time F./Min. 300/10 300/10 300/10 300/10 300/10 300/10 300/10 300/10 300/10 300/10 300/10 Softener Amt %OMF 1.5 5. 5. 1.5 1.5 15 1,5 5 5. 5 5. Fabric CH<sub>2</sub>O Cet # 9 Softener New Cherokke %OWF %OWF Type Softener Type ELS ELS E.S ELS ELS 皇 ELS 皇 9 모 유 Cet # 9 2.0 2 80 0 80 0 8.0 0 5.0 0.8 5 30 3.0 3.0 5.0 20 9.0 50 3.0 3.0 30 Fabric New Cherokee Oxford Shirting 100% Cotton 100% Corton 100% Cotton 100% Cotton 100% Cotton 100% Certon 100% Cotton 100% Cotton 100% Cotton 100% Cotton 100% Cotton Example No. = 12 <del>1</del>3 5 9 1 € 49 ೭ 7 F

	_	_		$\overline{}$	ì	
	22 100% Cofficial 50 20 HD 15 300/10 33.2 x 3.5 26 8 x 210 108 x 0 92 300 108 x 100 315 26.3 x 410 17 6 x 19 9			741X1087 774X1038 292X187 <10 330X100 <10 701X1097 377X594		
į	263×410			701 X 109 7		
	3 15			<10		
	108 X 100			330 X 100	:	
	3 00			<10		
nued)	1.08 × 0.92			2 92 X 1 67		
TABLE NO. II (Continued)	266X210			77.4 X 103.B	ashing.	
TABLE NO	33.2 X 32.5			74.1 X 108.7	Evaluated after treatment but before washing.	
	300/10			!	nt but	ngs.
	1.5			1	atme	vashir
	유			!	ter tre	Evaluated after 5 washings.
	2.0			1	ed af	ted af
	5.0			!	aluat	alua
	100% Cotton			100% Coffor	<u>.</u> 厄	с: Ш
	22		Washed Control (5Washe s)		<b>~</b>	(1

#### What is claimed is:

1. A durable press process for cellulosic fiber-containing fabrics comprising treating a cellulose fiber-containing fabric with formaldehyde, a catalyst capable of catalyzing the crosslinking reaction between formaldehyde and cellulose, and an effective amount of silicone elastomer, heat curing said treated cellulosic fiber-containing fabric under conditions at which formaldehyde reacts with cellulose in the presence of the catalyst and silicone elastomer, without a substantial loss of formaldehyde before the reaction of the formaldehyde with the cellulose to improve the wrinkle resistance of the fabric while reducing loss in tear and tensile strength.

15

10

5

2. The process of claim 1 wherein the heat curing is at a temperature which prevents the substantial loss of formaldehyde during curing.

20

3. The process of claim 1 wherein the heat curing step is carried out at a high enough temperature to allow the crosslinking step to occur before sufficient formaldehyde leaves the fabric and affects the process.

- 4. The process of claim 1 wherein the fabric being cured has a moisture content of more than 20% by weight.
- 5. The process of claim 2 wherein the heat curing is over a temperature of from 100°F to 350°F.

6. The process in claim 2 where the heat curing is carried out in the preferred range of 250 to 325°F.

- 7. The process of claim 1 wherein said fabric is heat cured by gradually increasing the temperature.
  - 8. The process of claim 5 wherein the heat curing is over a temperature of from 100°F to 300°F.
- 10 9. The process in claim 1 where the formaldehyde is in the form of an aqueous solution of formaldehye having a concentration of 0.5% to 10%.
  - 10. The process in claim 1 where the preferred formaldehyde concentration range is from 1.5% to 7% on the weight of the fabric.
    - 11. The process of claim 1, wherein the fabric is 100% cotton shirting.

20

15

#### INTERNATIONAL SEARCH REPORT

International application No. PCT/US98/09367

IPC(6) US CL	ASSIFICATION OF SUBJECT MATTER :D06M 13/127 :8/116.4, 115.7; 427/389.9, 394 to International Patent Classification (IPC) or to both	national classification and IPC	
· · · · · · · · · · · · · · · · · · ·	LDS SEARCHED		
Minimum o	documentation searched (classification system follows	ed by classification symbols)	
U.S. :	8/116.4, 115.7; 427/389.9, 394		
Documenta	tion searched other than minimum documentation to the	ne extent that such documents are included	in the fields searched
Electronic (	data base consulted during the international search (n	name of data base and, where practicable	, search terms used)
C. DOC	CUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where a	ppropriate, of the relevant passages	Relevant to claim No.
X	US 3,663,974 A (WANTANABE et a 8-44; and examples 1-3 and 12.	al) 23 May 1972, col. 2, lines	1-3, 5, 8-11
Y	,		4, 6, 7
A	US 2,243,765 A (MORTON) 27 May	1941, col. 1, lines 1-32.	1-11
A	US 3,812,201 A (BEY) 21 May 1974	, col. 1, lines 10-14.	1-11
A	US 4,108,598 A (PAYET) 22 August	1978, col. 2, lines 23-33.	1-11
A	US 4,269,603 A (WORTH) 26 May 1	1981, col. 2, lines 21-32.	1-11
X Furth	er documents are listed in the continuation of Box C	See patent family annex.	
	ecial categories of cited documents: cument defining the general state of the art which is not considered	"T" later document published after the inte date and not in conflict with the appli	ication but cited to understand
to	be of particular relevance  lier document published on or after the international filing date.	the principle or theory underlying the  'X' document of particular relevance; the	
"L" do	cument which may throw doubts on priority claim(s) or which is ed to establish the publication date of another citation or other	considered novel or cannot be consider when the document is taken alone	ed to involve an inventive step
*C* doc	scial reason (as specified)  cument referring to an oral disclosure, use, exhibition or other	"Y" document of particular relevance; the considered to involve an inventive combined with one or more other such	step when the document is documents, such combination
*P* dox	ans cument published prior to the international filing date but later than priority date claimed	being obvious to a person skilled in the "&" document member of the same patent	
	actual completion of the international search	Date of mailing of the internal 1998 a	rch report
19 JUNE	1998	10 AUG 1930	
Commission Box PCT	nailing address of the ISA/US ner of Patents and Trademarks n, D.C. 20231	Authorized afficer  ALAN D. DIAMOND	
Facsimile N	o. (703) 305-3230	Telephone No. (703) 308-0661	

#### INTERNATIONAL SEARCH REPORT

International application No.
PCT/US98/09367

		FC1/0398/0930	•
C (Continua	tion). DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relev	ant passages	Relevant to claim No
A	US 4,369,390 A (HENDRIX et al) 02 August 1983, co 10.	l. 2, lines 1-	1-11
		:	
:			
:			
		:	